

National Aeronautics and Space Administration

Modeling of Different Fiber Type and Content SiC_f/SiC Minicomposites Creep Behavior

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Outline

- **Motivation**
- **Approach**
- **Materials and Properties**
- **Experimental Methodology and Set Up**
- **Creep Testing Results**
- **Constituents' Creep Characterization**
- **Creep Model**
- **Conclusions**

Motivation

- **Ceramic Matrix Composites (CMCs)** are candidates for high-temperature applications such as the new generations of aircraft engines and hypersonic vehicles.
- **Case A**: CMCs are loaded below the matrix cracking stress. (Static or Dynamic Loading)
 - Fibers carry a fraction of the applied load.
 - Fibers are not exposed to oxidation damage and the most dominant damage mechanism is creep of fibers.
- **Case B**: CMCs are loaded above the matrix cracking stress.
 - Fibers carry all of the applied load in the vicinity of through-thickness matrix crack.
 - Fibers are exposed to oxidation and creep damage mechanisms.
 - Load transfer from oxide layer to the core of the fibers.
 - Long lengths of fibers at high stress due to multiple matrix cracks and the reduction of fibers' cross-sectional area due to oxidation.

Approach

1) Case A:

Obtain Fibers true creep parameters at 1200-1482 ° C which are needed for modeling fiber/matrix load transfer and creep resistance of the fibers.

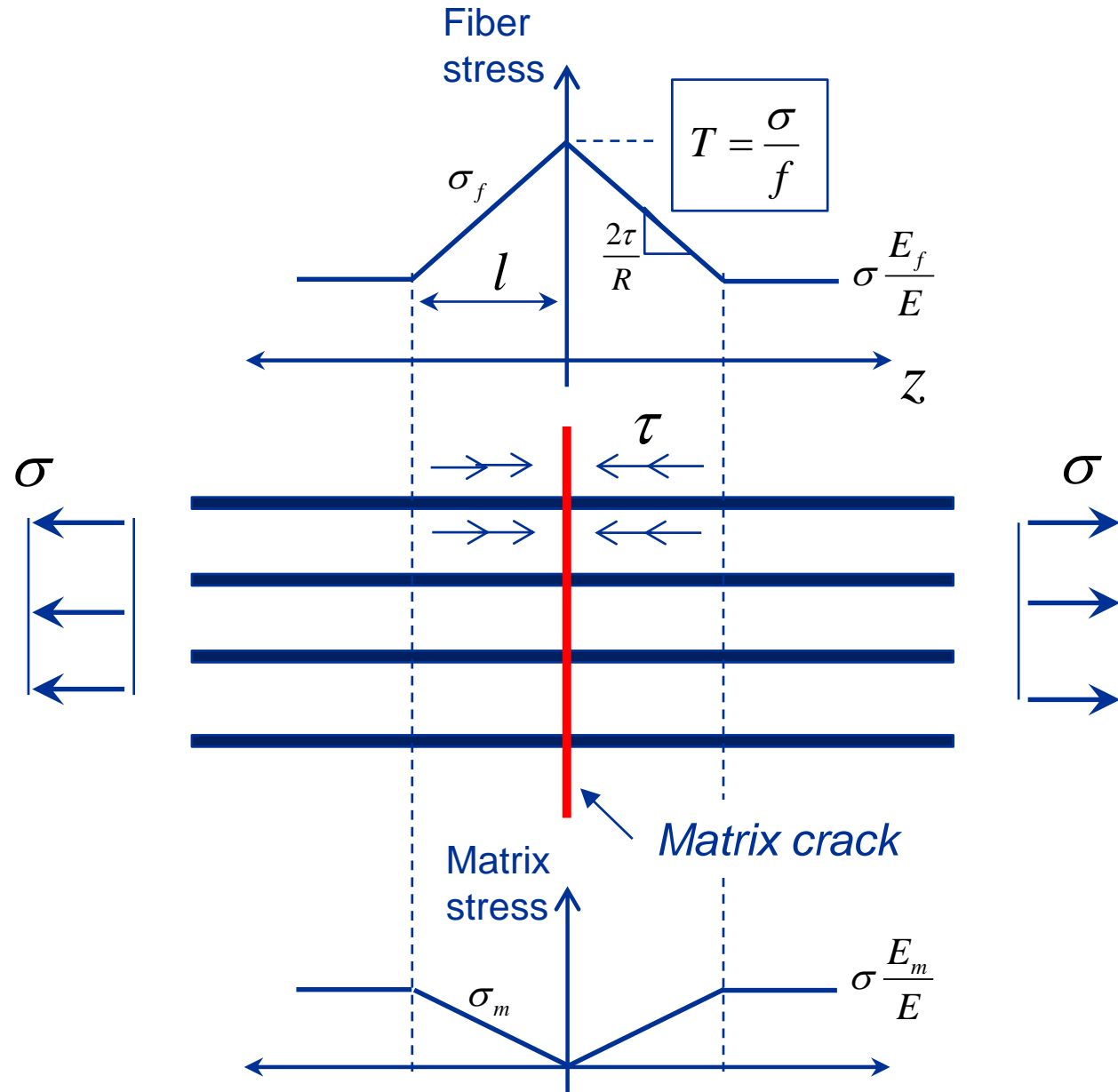
- A) Test single fibers and fiber tows in vacuum.
- B) Test single fibers and fiber tows in air (*need to model the evolution of stress increase on fibers during creep due to oxidation*).

Case B:

Obtain Fibers creep parameters at 1200-1482 ° C tows in air, inert and steam.

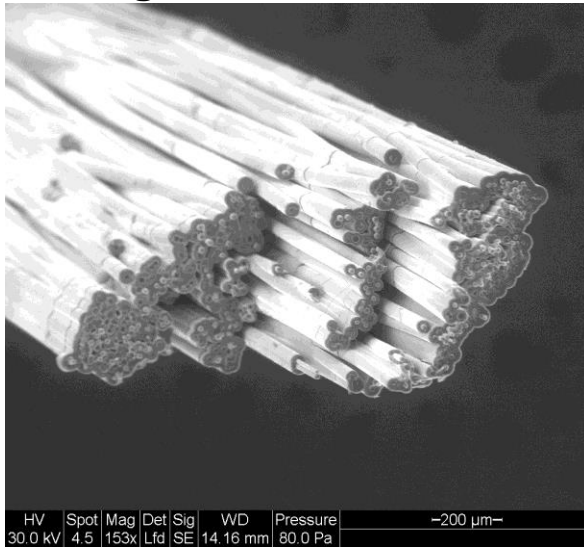
2) Correlate fibers data with minicomposites and macrocomposites data.

3) Provide recommendation for durability improvements & Support CMCs & fibers developments.

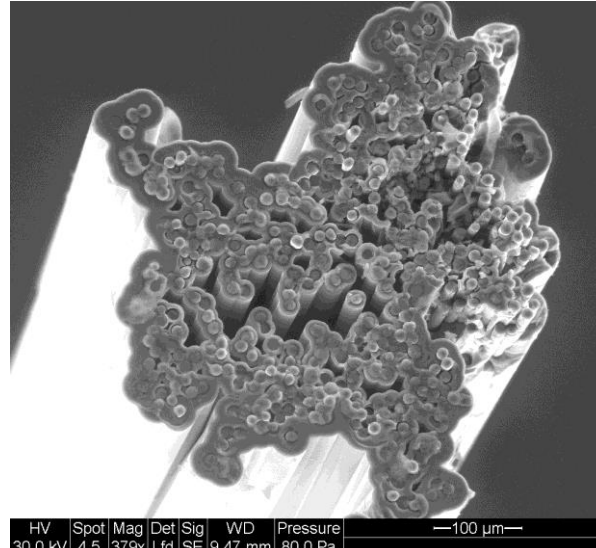


Materials and Properties

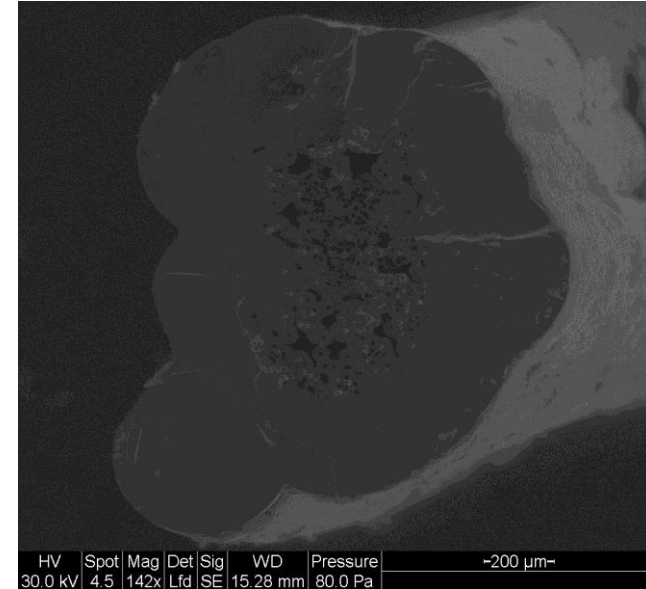
Single Fiber Tow Minicomposite Cross-sections



Hi-Nicalon Minicomposite



Hi-Nicalon S Minicomposite

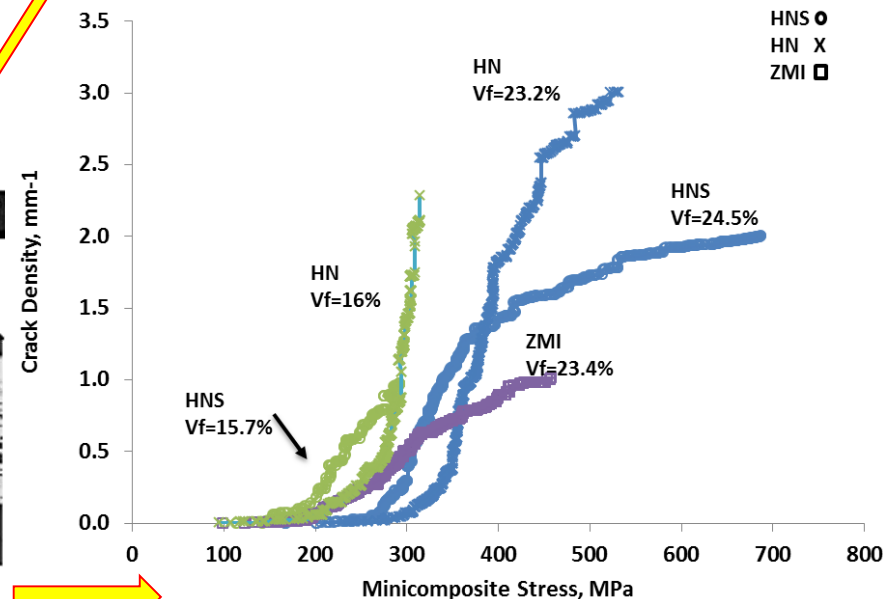
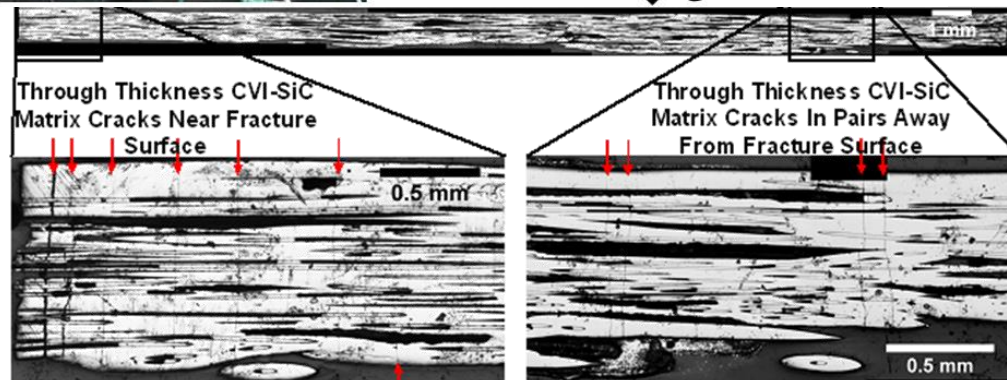
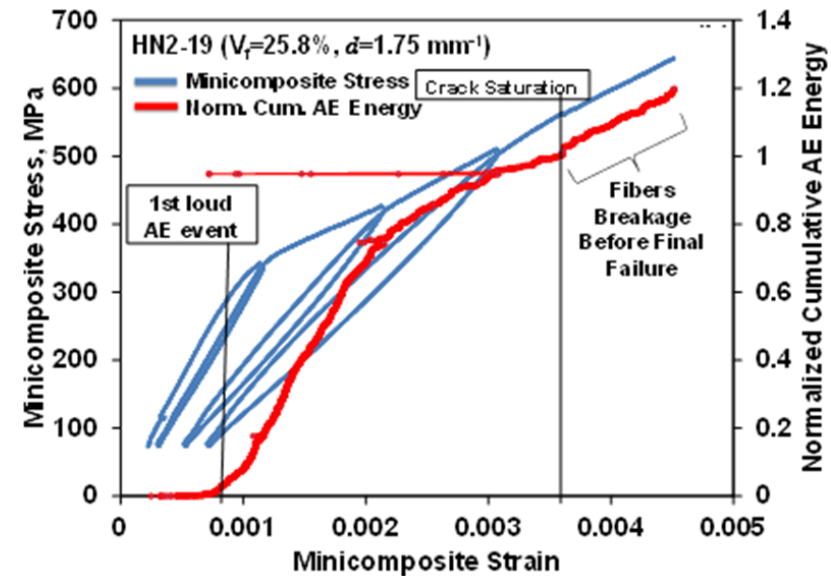
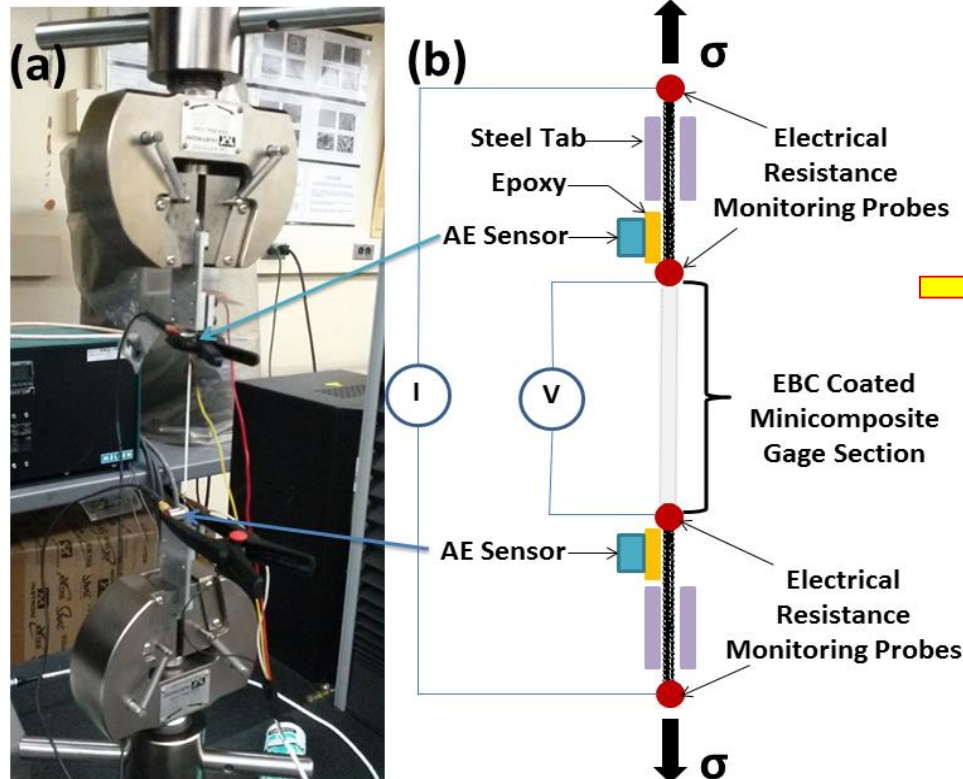


97%CVI-SiC Minicomposite

Fiber Type	Number of Fibers per Tow	Fiber Diameter (μm)	Fiber Volume Fractions (%)	Fiber Elastic Modulus (GPa)	Fiber Density (g/cc)	Fiber Coefficient of Thermal Expansion (CTE)(°C ⁻¹)
Hi-Nicalon-S	500	12	16/23/43	400	3.1	4.5×10^{-6}
Hi-Nicalon	500	14	16/23/42	270	2.74	3.5×10^{-6}
Tyranno-ZMI	400	11	23/28	170	2.35	4.0×10^{-6}

- The volume and mass of the fiber tow were estimated based on the average fiber diameter, number of fibers per tow, specimen length and density.
- The volume and mass of the interphase were estimated considering a constant thickness of 1 μm on each fiber using SEM.
- Then backed out the volume and mass of the matrix from ROM.

Precracking Minis Methodology at Room Temperature



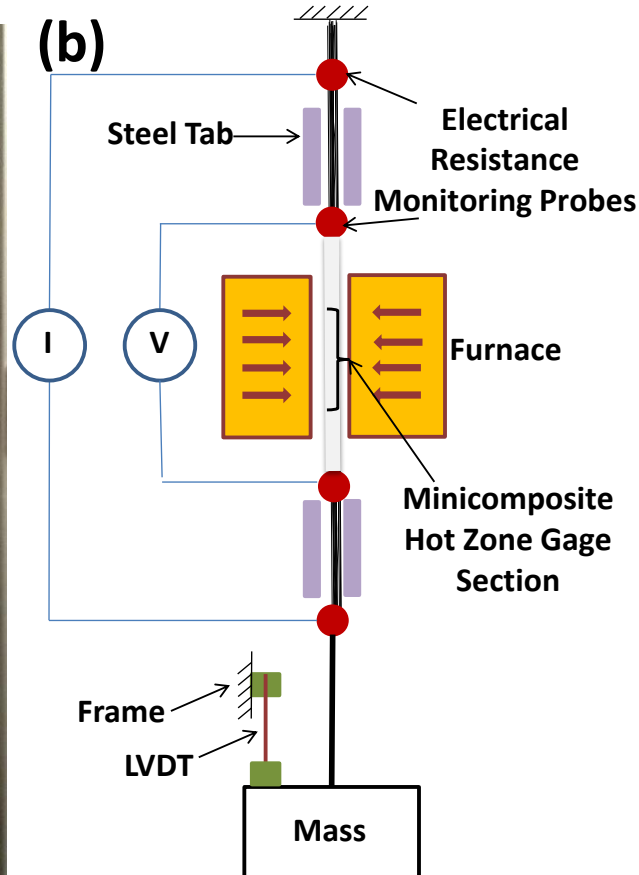
High Temperature Creep Test Setup in Air

Minicomposites Creep Test Rig

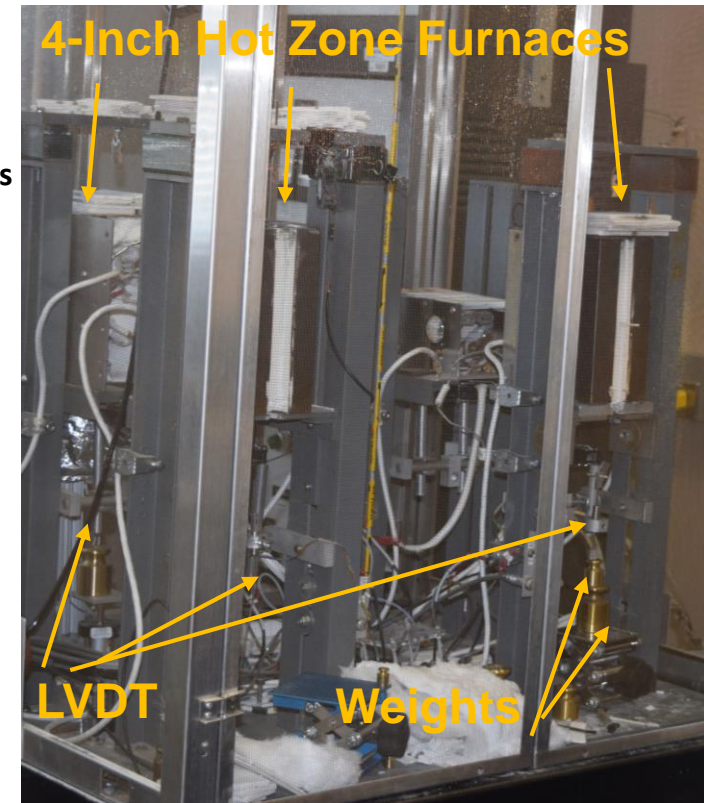
(a)



(b)

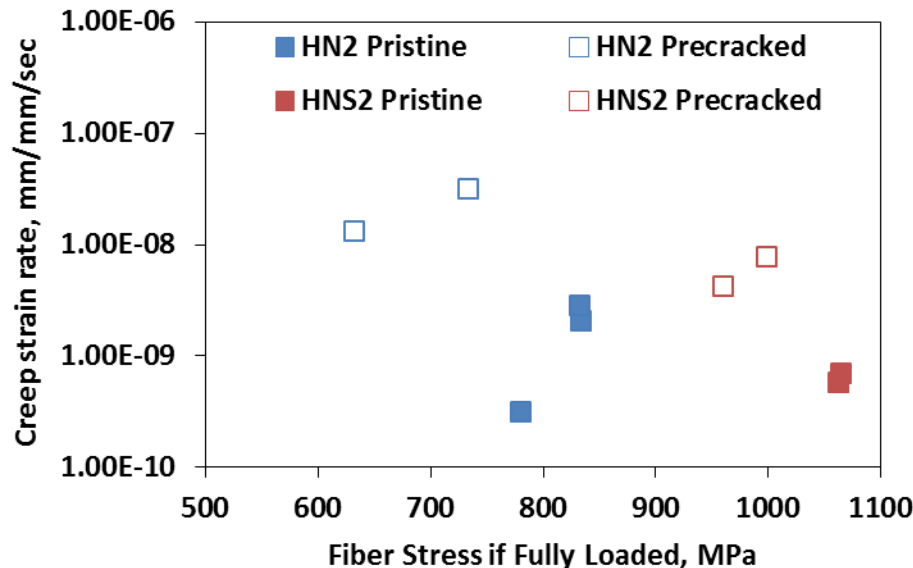


Single Fiber Creep Test Rig

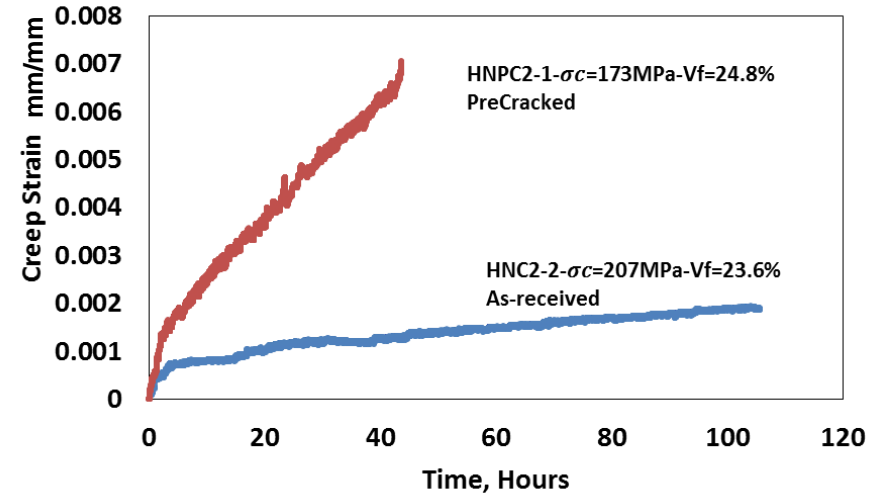


Uncracked Vs. Cracked Minicomposites' Creep Behavior

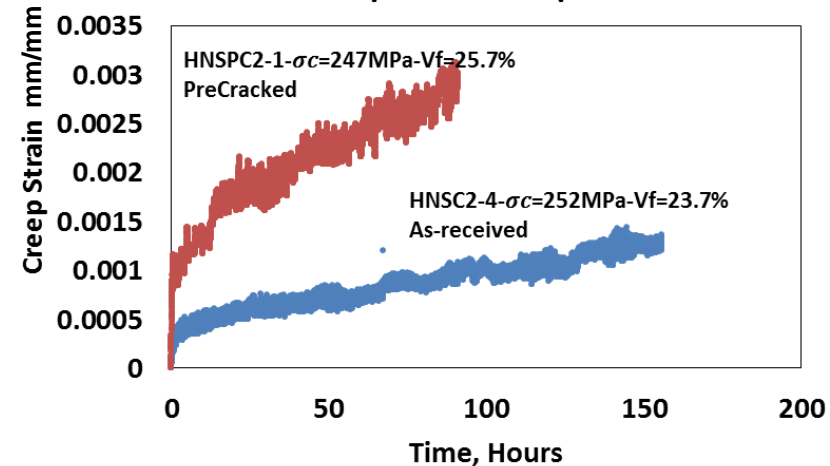
- Larger total strain and strain rates in Precracked HN and HNS due to longer lengths of fibers at higher stress and oxidation
- Smaller total strain in As-Produced HN and HNS due to load sharing (matrix carries some load – fiber stress isn't as high as for the precracked).
- Precracked samples failed earlier than the pristine samples under the same loading conditions.



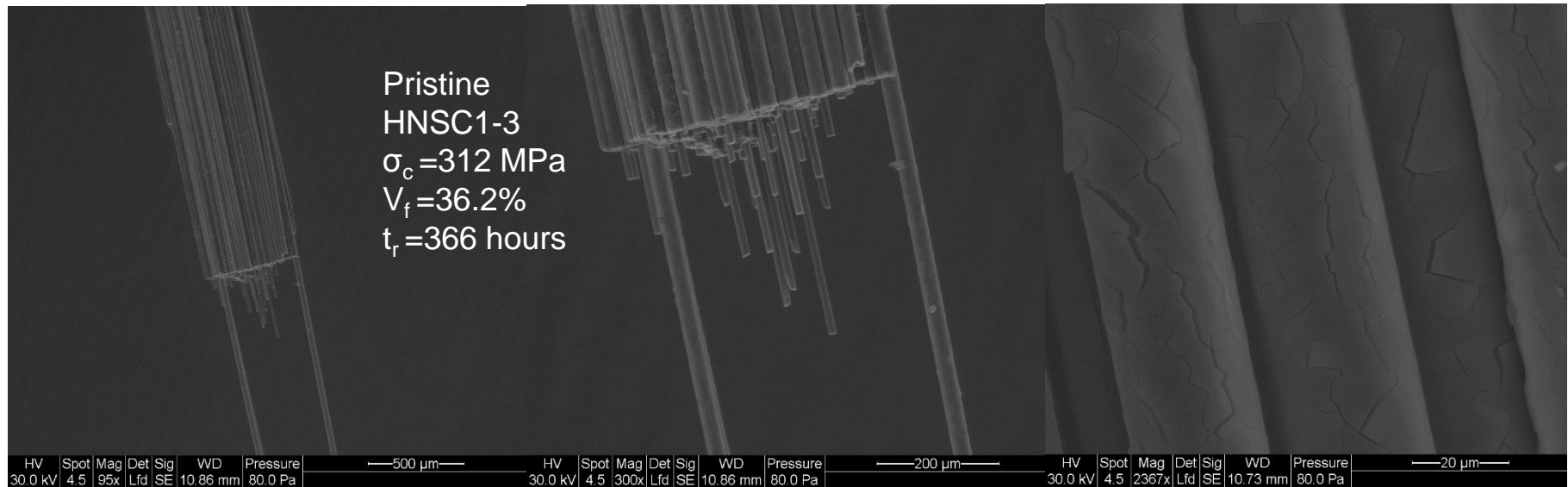
As-received and PreCracked Hi-Nicalon minicomposites Creep Curves



As-received and PreCracked Hi-Nicalon Type S Minicomposites Creep Curves



Post Creep Fracture Surface Morphology

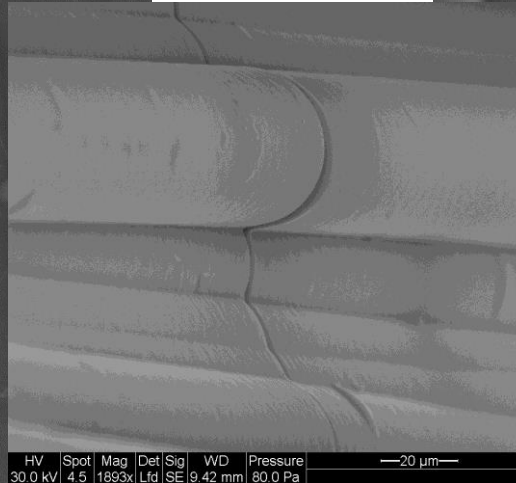


- Obvious fiber pull-out in **pristine** HNSC1-3 sample tested in creep.
- Also, **cracked oxide** layer on the surface of the pristine sample due to the long time it lasted in creep, thermal cycle and the surrounding air.
- Little to no fiber pull-out (**brittle surface**) due to fiber creep and oxidation degradation in **precracked** sample HNSPC2-1.

High Temperature Oxidation Behavior

B 5.49 C 11.21
O 44.42 Si 37.49

B 6.7 C 11.78
O 29.49 Si 50.74



HNSPC2-4
Side Crack

**Precracked
HNSPC2-4**
 $\sigma_c = 201 \text{ MPa}$
 $V_f = 23.3\%$
 $t_r = 516 \text{ hours}$

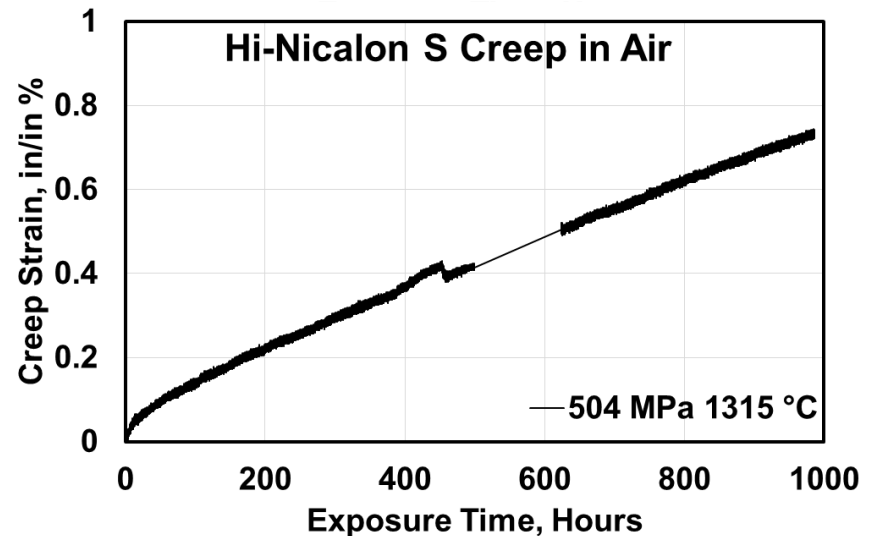
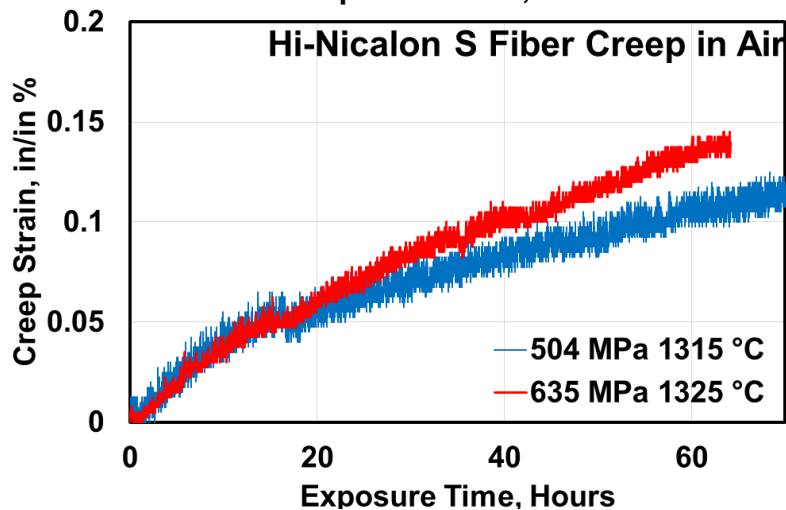
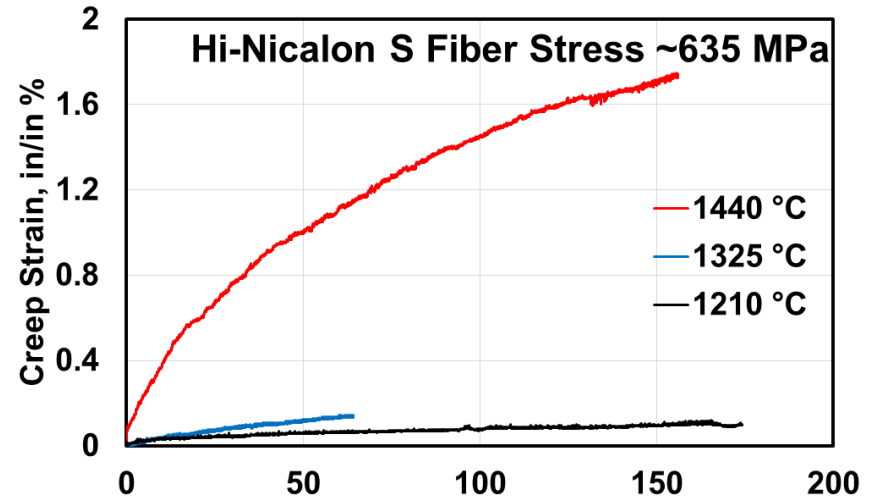
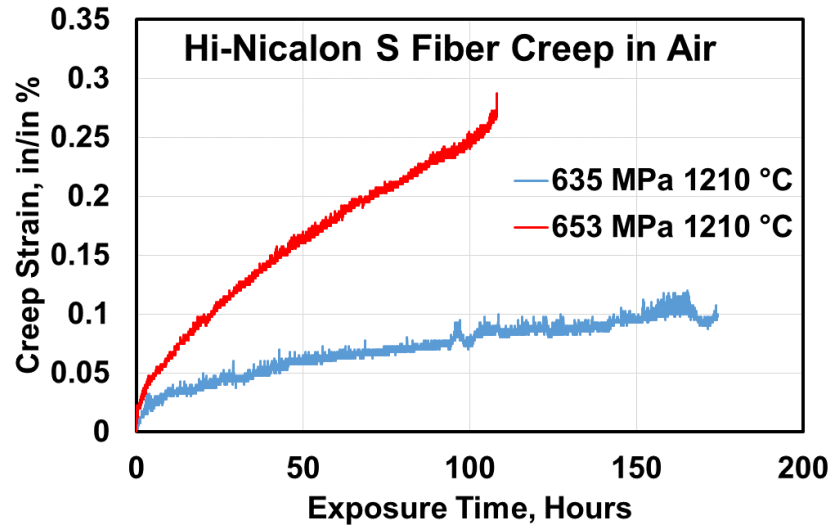
HNSPC1-2
Side Crack

**Precracked
HNSPC1-2**
 $\sigma_c = 344 \text{ MPa}$
 $V_f = 34.5\%$
 $t_r = 8 \text{ hours}$

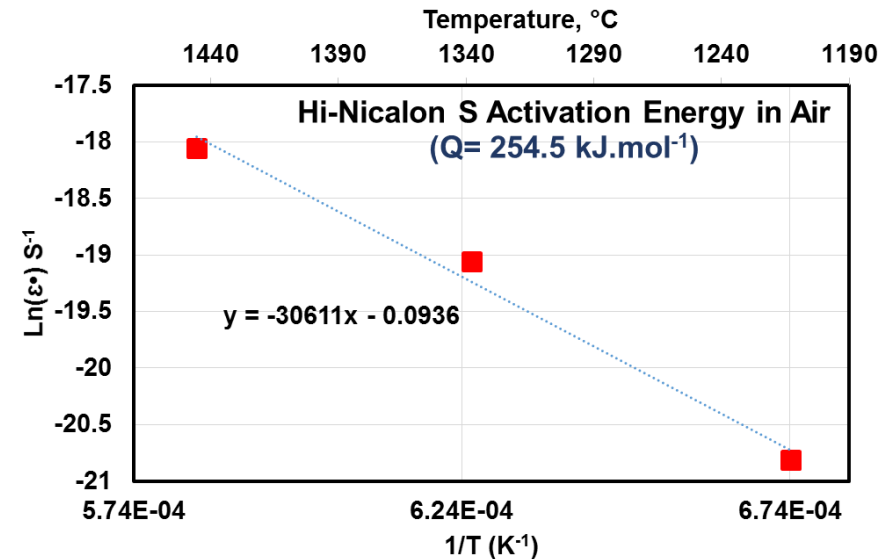
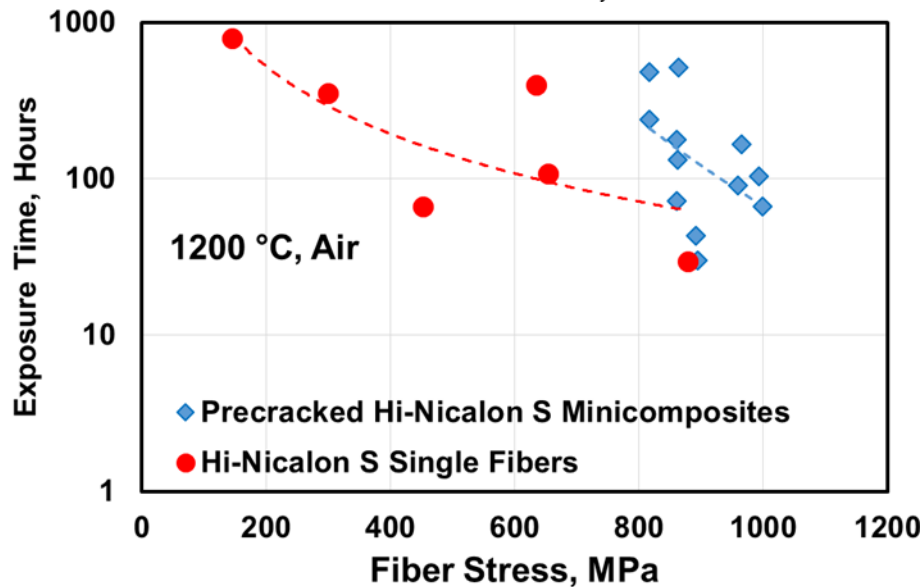
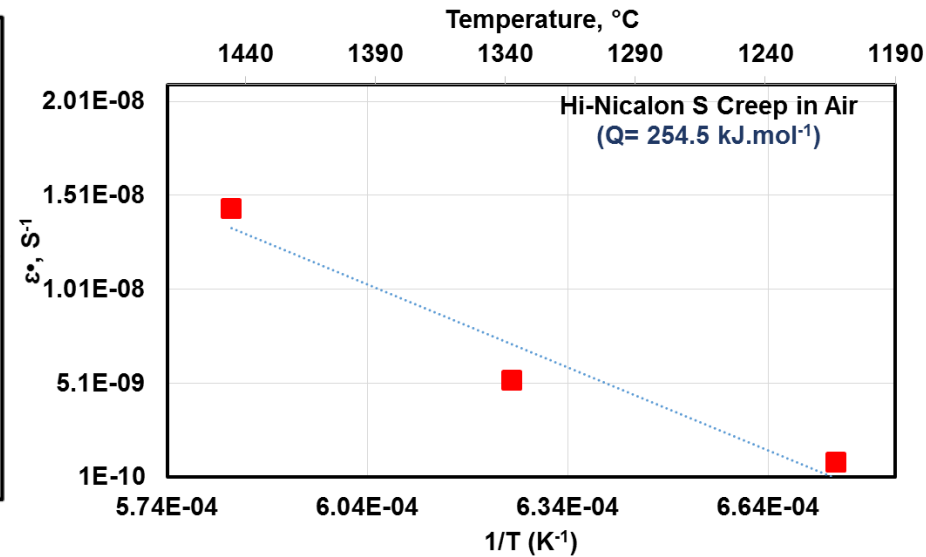
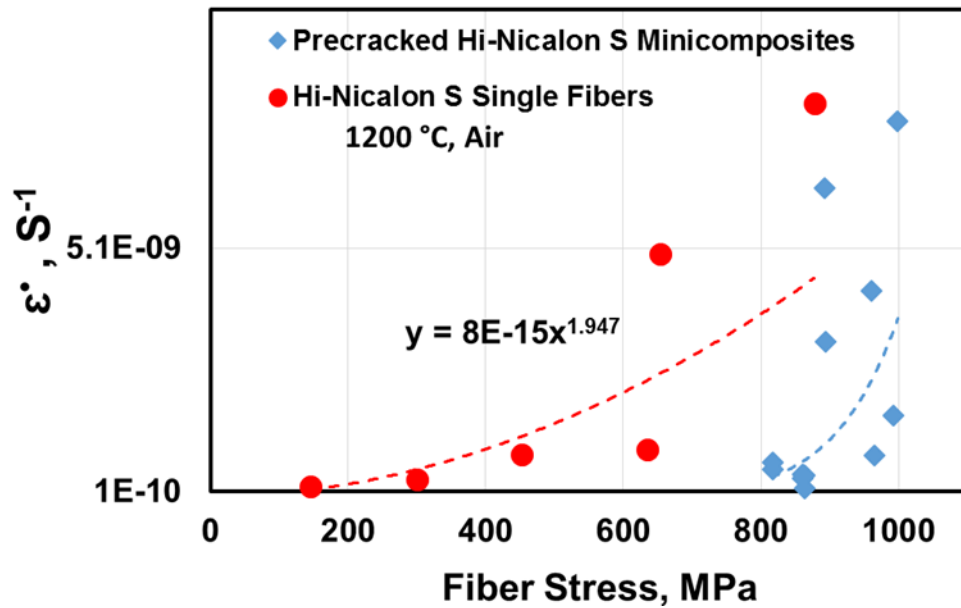
HV	Spot	Mag	Det	Sig	WD	Pressure
30.0 kV	5.0	1420x	Lfd	SE	9.93 mm	80.0 Pa

HV	Spot	Mag	Det	Sig	WD	Pressure
30.0 kV	5.0	2367x	Lfd	SE	10.19 mm	80.0 Pa

Hi-Nicalon S Single Fiber Creep in Air



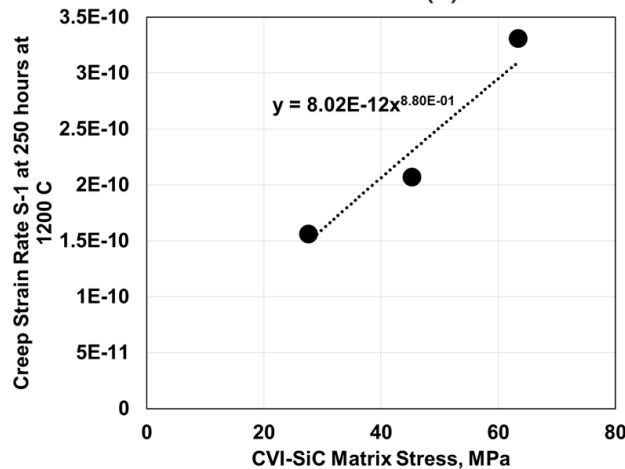
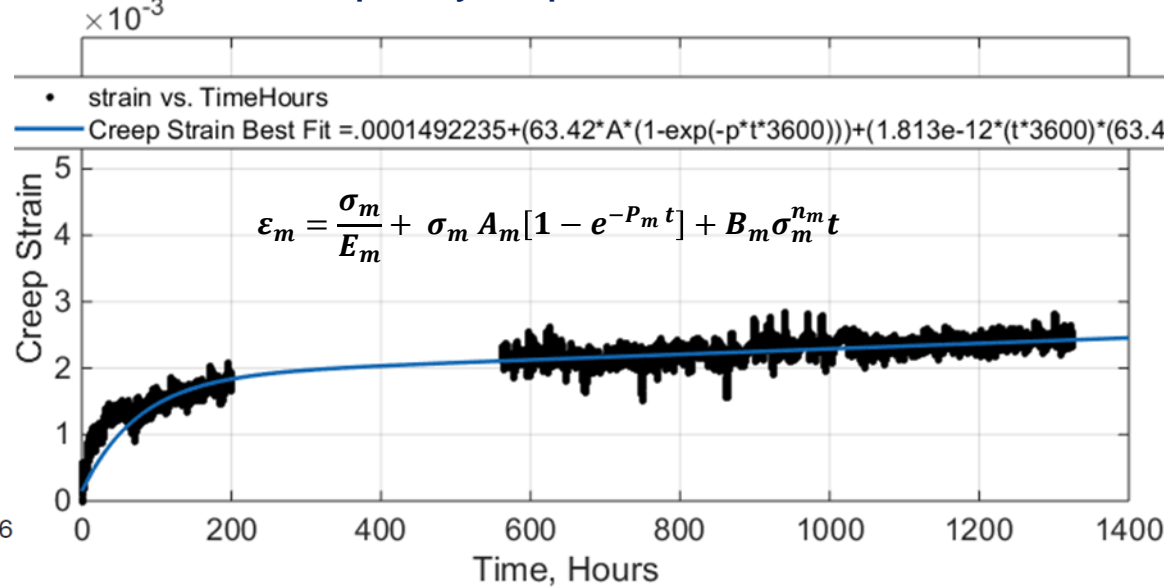
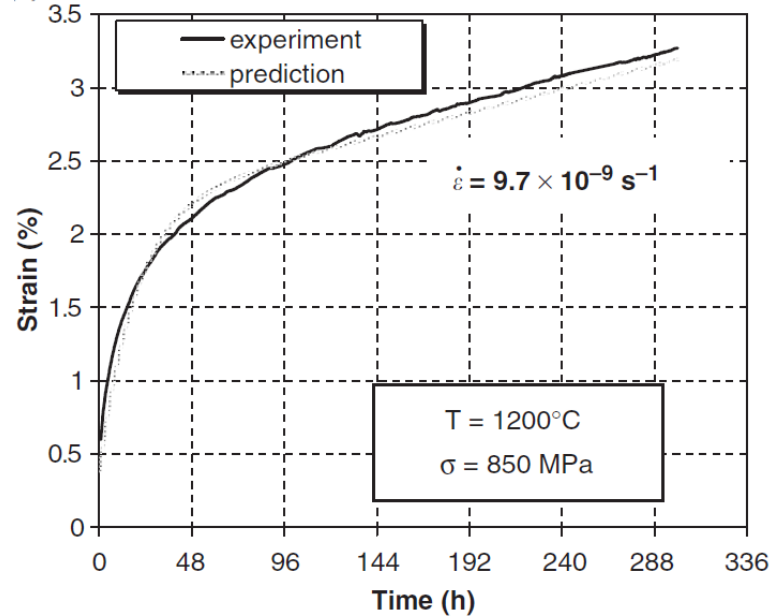
Hi-Nicalon S Single Fiber Creep in Air



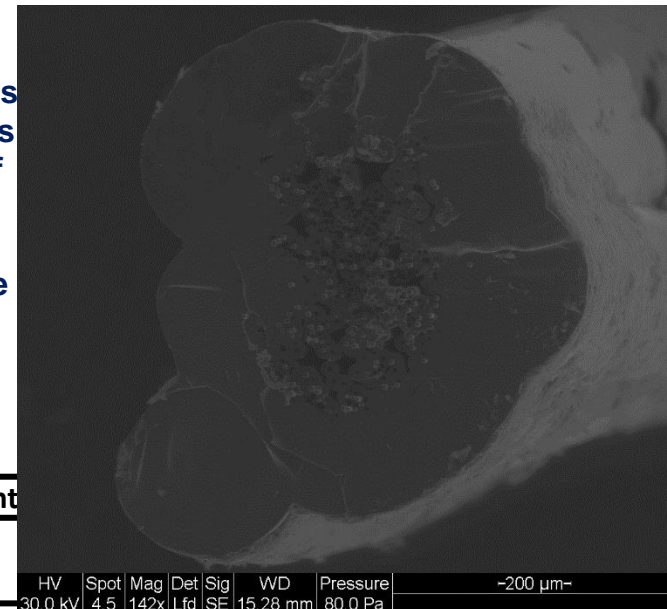
Fibers and Matrix Creep Properties at 1200 °C

Hi-Nicalon fibers primary and steady state parameters obtained from *Sauder&Lamon 2007*

CVI-SiC matrix creep curve that lasted for more than 1350 hours in creep at 1200 C was best fitted using Matlab curve fitting application to obtain CVI-SiC primary creep constants.



- CVI-SiC matrix steady state properties obtained from plotting 3 CVI-SiC tests strain rates at 1200 C as a function of stress.
- 97% CVI-SiC matrix with 3% HNS fibers Minis were assumed to be pure CVI-SiC



Constituent	A	p	B (s ⁻¹ /MPa)	n (Stress exponent)
Hi-Nicalon Fibers	2.127×10^{-5}	1.485×10^{-5}	1.77×10^{-15}	2.3
CVI-SiC Matrix	2.59×10^{-5}	4.432×10^{-6}	1.81×10^{-12}	1

Creep Stress Transfer Model

$$\varepsilon_f = \frac{\sigma_f}{E_f} + \sigma_f A_f [1 - e^{-P_f t}] + B_f \sigma_f^{n_f} t$$

$$\varepsilon_m = \frac{\sigma_m}{E_m} + \sigma_m A_m [1 - e^{-P_m t}] + B_m \sigma_m^{n_m} t$$

$$\dot{\varepsilon}_f = \frac{\dot{\sigma}_f}{E_f} + \dot{\sigma}_f A_f - \dot{\sigma}_f A_f e^{(-P_f t)} + \sigma_f A_f P_f [e^{(-P_f t)}] + B_f \sigma_f^{n_f}$$

$$\dot{\varepsilon}_m = \frac{\dot{\sigma}_m}{E_m} + \dot{\sigma}_m A_m - \dot{\sigma}_m A_m e^{(-P_m t)} + \sigma_m A_m P_m [e^{(-P_m t)}] + B_m \sigma_m^{n_m}$$

$$\dot{\sigma}_m = \frac{\sigma - \sigma_f V_f}{V_m}$$

$$\sigma = 0$$

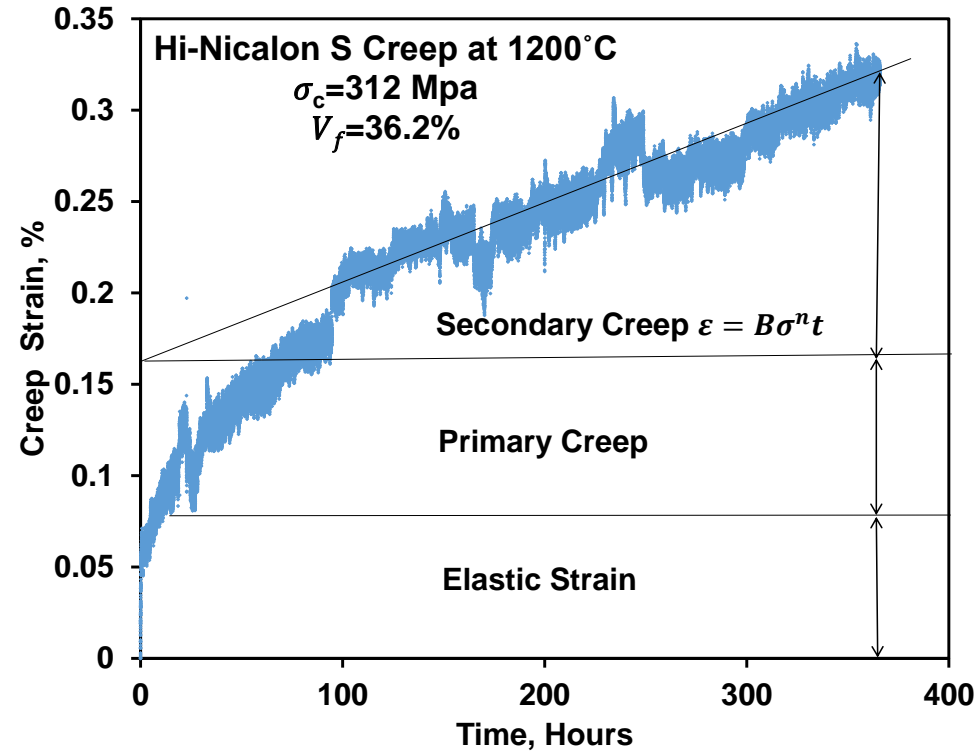
$$\varepsilon_f = \varepsilon_m$$

$$\dot{\varepsilon}_f = \dot{\varepsilon}_m$$

$$\dot{\sigma}_f = \frac{\sigma_m A_m P_m [e^{(-P_m t)}] - \sigma_f A_f P_f [e^{(-P_f t)}] + B_m \sigma_m^{n_m} - B_f \sigma_f^{n_f}}{\left[\frac{1}{E_f} + A_f - A_f e^{(-P_f t)} + \frac{V_f}{V_m E_m} + \frac{A_m V_f}{V_m} - \frac{A_m V_f e^{(-P_m t)}}{V_m} - B_f n_f \sigma_f^{n_f-1} t - \frac{B_m n_m V_f \sigma_m^{n_m-1} t}{V_m} \right]}$$

$$\sigma_{f(i)} = \sigma_{f(i-1)} + \Delta t \dot{\sigma}_{f(i-1)}; \Delta t = t_i - t_{i-1}$$

$$\sigma_{m(i)} = \sigma_{m(i-1)} + \Delta t \dot{\sigma}_{m(i-1)}; \Delta t = t_i - t_{i-1}$$



Δt is 10 seconds in the model
 Stress on the fibers and the matrix increased with the increase in V_f .

Creep Model Illustration

$$\varepsilon_f(t_i) - \frac{\sigma_f}{E_f} - \sigma_f A_f [1 - e^{-P_f t_i}] - B_f \sigma_f^{n_f} t_i = 0$$

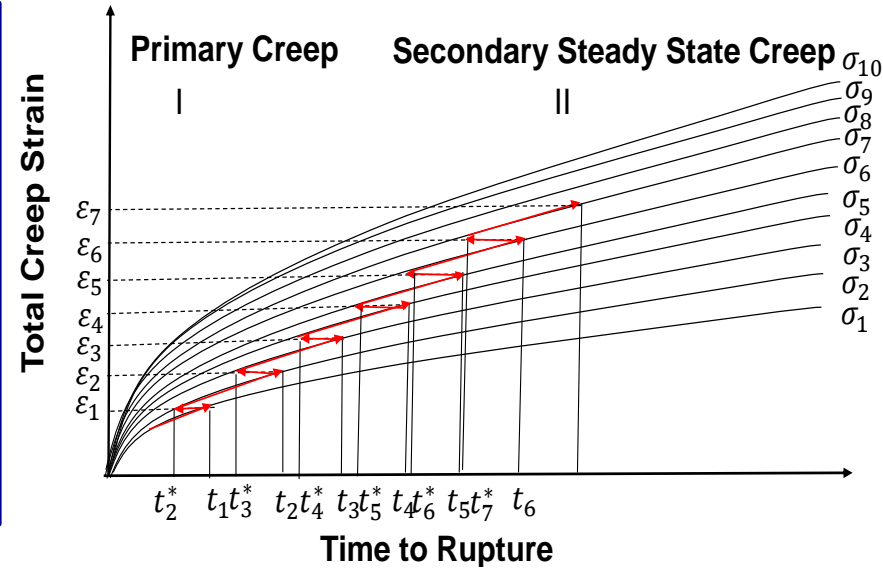
$$t_{i+1}^* = t_i - \frac{\varepsilon_f(t_i)}{\varepsilon'_f(t_i)}$$

$$\varepsilon'_f(t_i) = \frac{\varepsilon_f(t_i) - \varepsilon_f(t_{i-1}^*)}{t_i - t_{i-1}^*}$$

$$t_{i+1}^* = t_i - \frac{\Delta * \varepsilon_f(t_i)}{\varepsilon_f(t_i) - \varepsilon_f(t_{i-1}^*)}$$

with $\Delta = t_i - t_{i-1}^* = 10^{-8}$ sec and Error $< 10^{-6}$

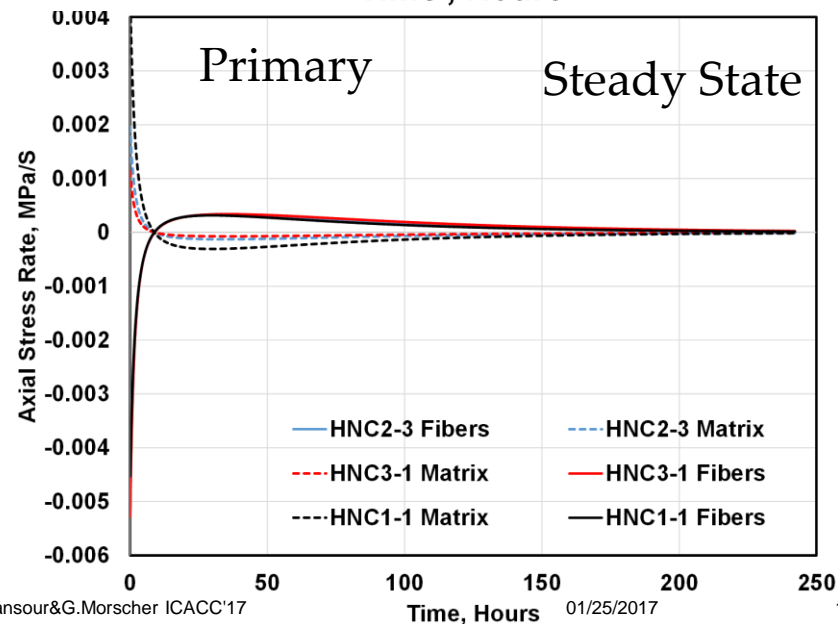
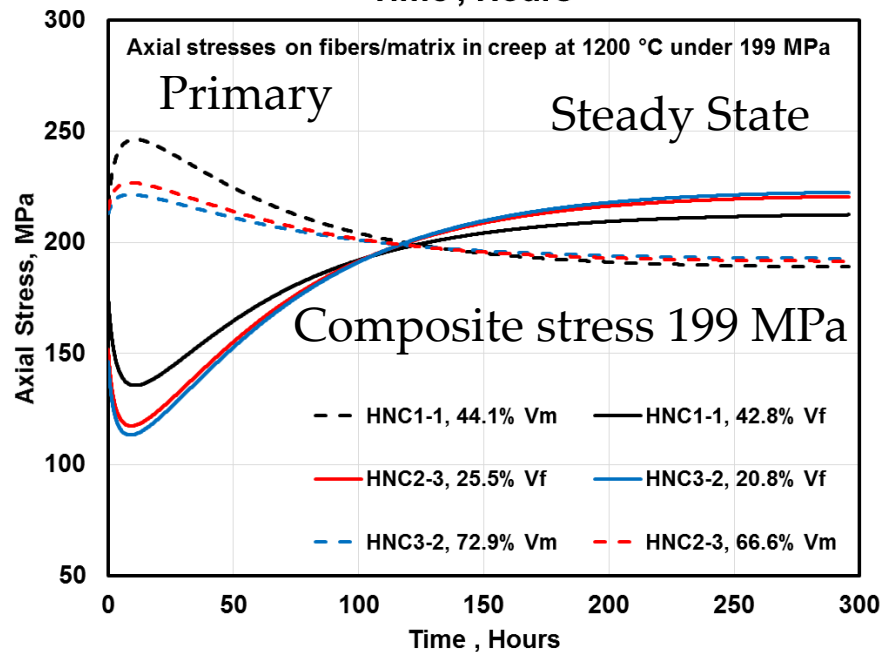
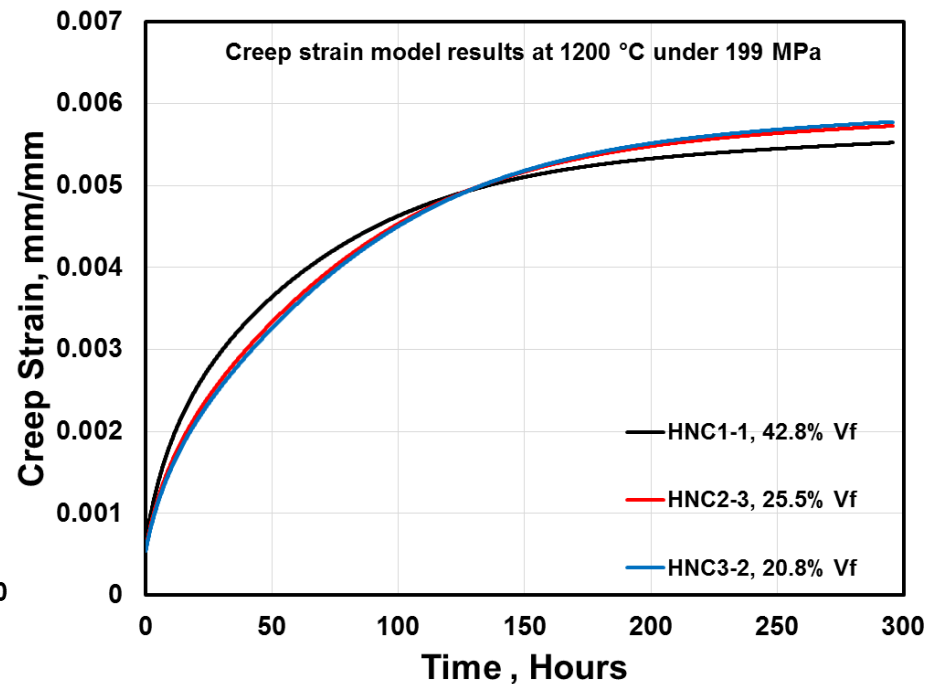
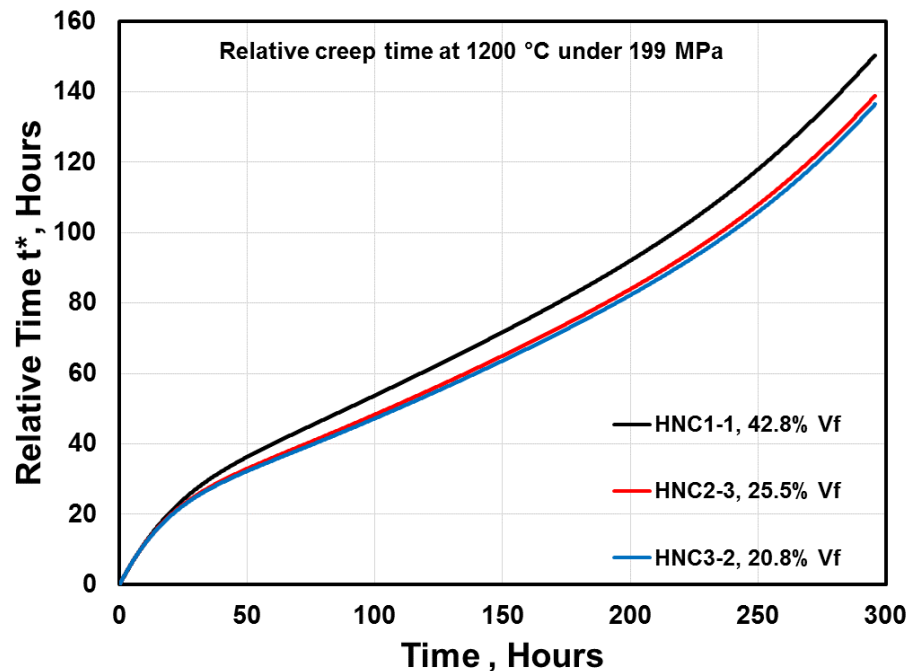
$$\varepsilon_{fi}(10 + t_{i+1}^*) = \frac{\sigma_{fi}}{E_f} + \sigma_{fi} A_f [1 - e^{-P_f (10 + t_{i+1}^*)}] + B_f \sigma_{fi}^{n_f} (10 + t_{i+1}^*)$$



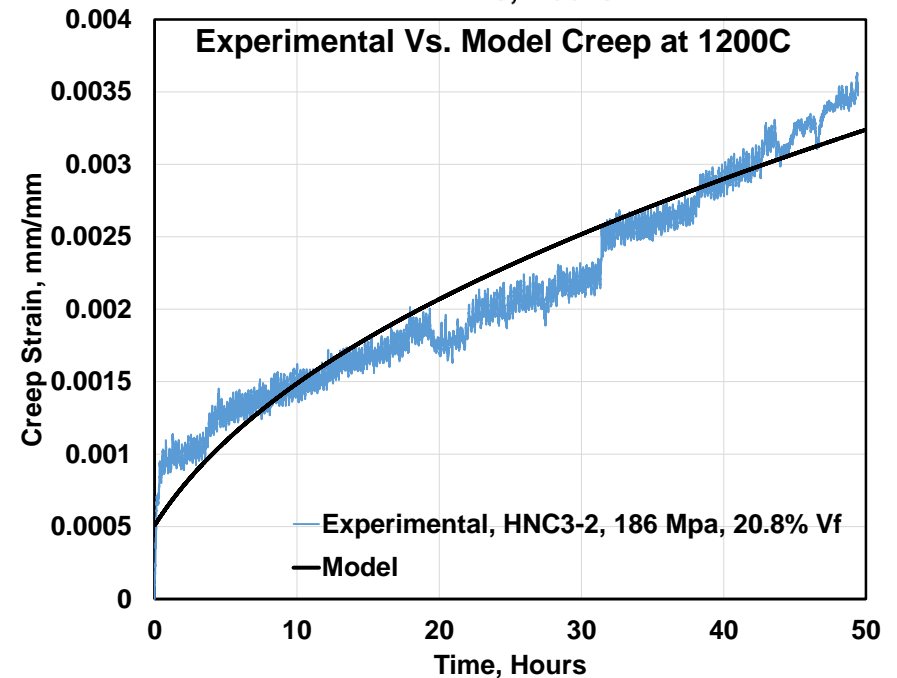
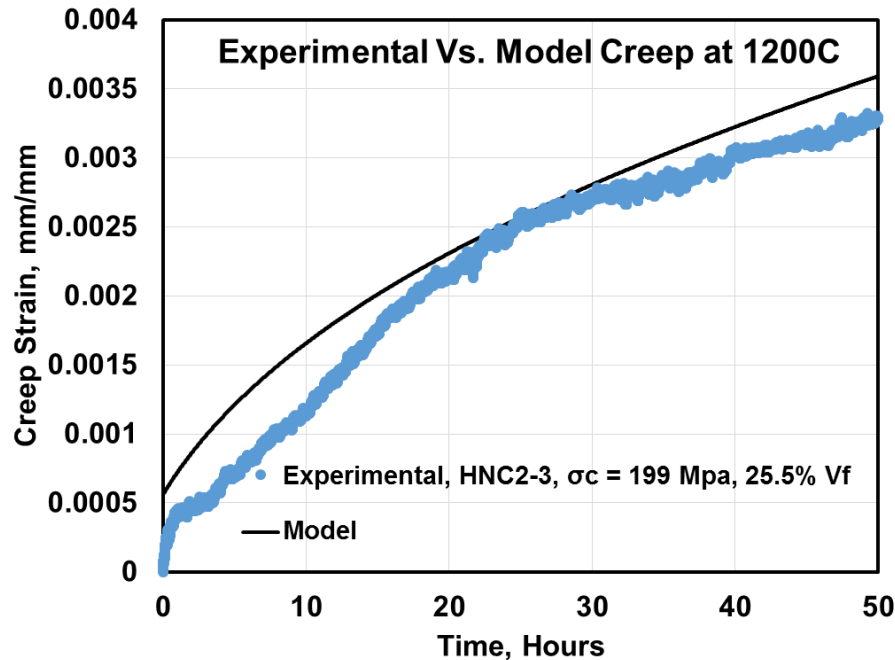
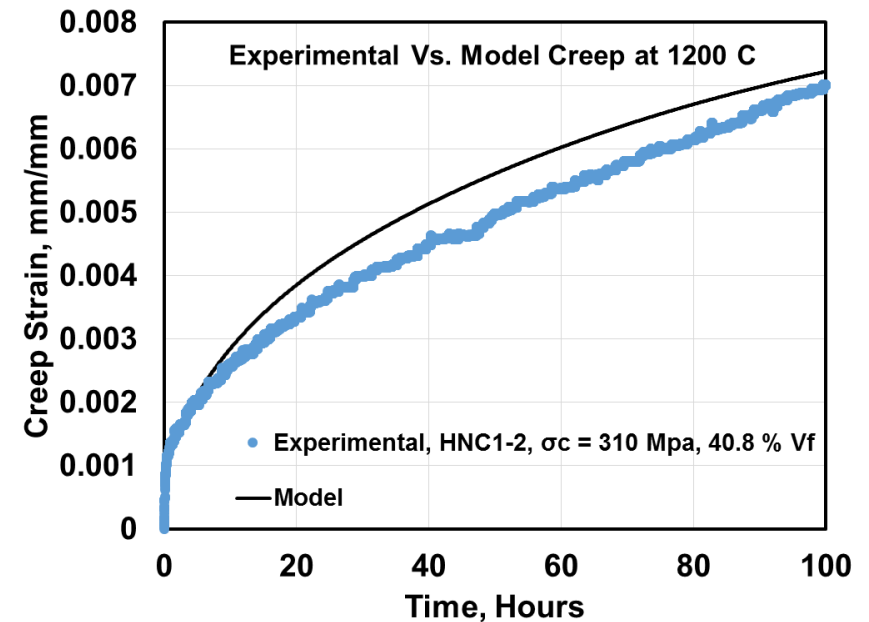
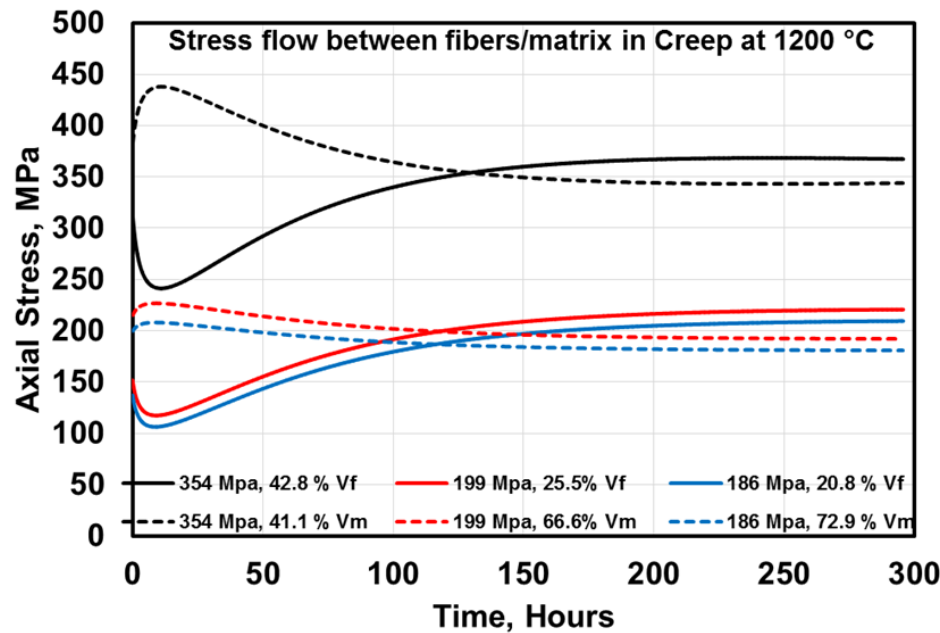
Real Time, t' , Seconds	Stress on Fibers, σ_{fi}	Fibers Strain, $\varepsilon_{i-1} (\sigma_{fi}, t'_i)$	Relative time, $t_i^* (\sigma_{fi}, \varepsilon_{i-1})$	Cumulative Time, $t_i = \sum t_i^*$	Fibers Creep Strain $\varepsilon_{fi}(\sigma_{fi}, t_{i+1}^* + 10)$
10	σ_1	ε_1	t_1^*	t_1	ε_{f1}
20	σ_2	ε_2	t_2^*	t_2	ε_{f2}
30	σ_3	ε_3	t_3^*	t_3	ε_{f3}
40	σ_4	ε_4	t_4^*	t_4	ε_{f4}
50	σ_5	ε_5	t_5^*	t_5	ε_{f5}
60	σ_6	ε_6	t_6^*	t_6	ε_{f6}

Where: $t' > t_n > t_n^*$
Time increment 10 Seconds

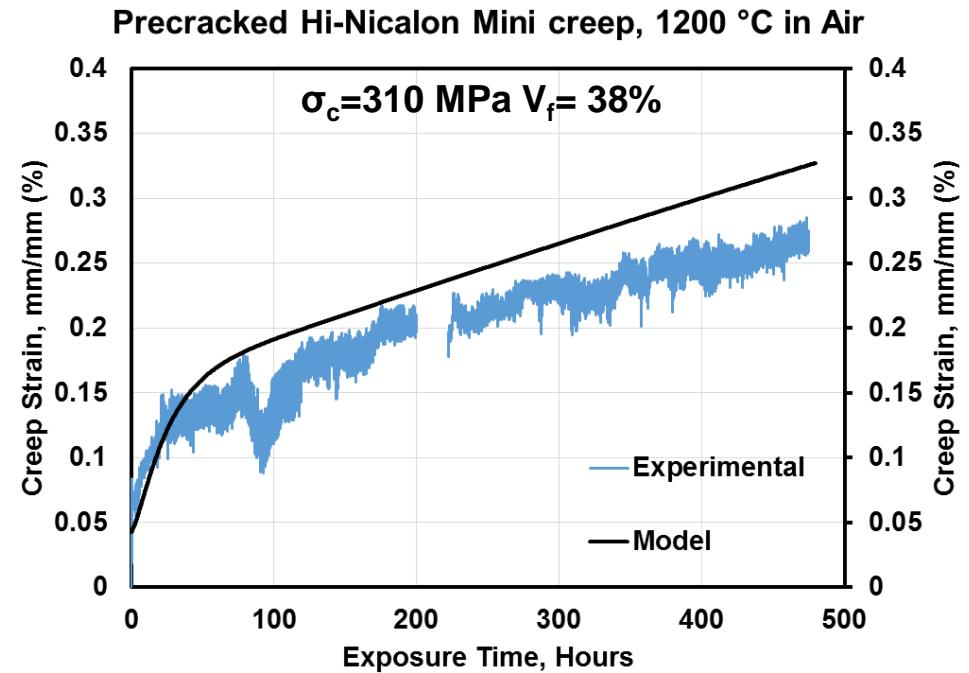
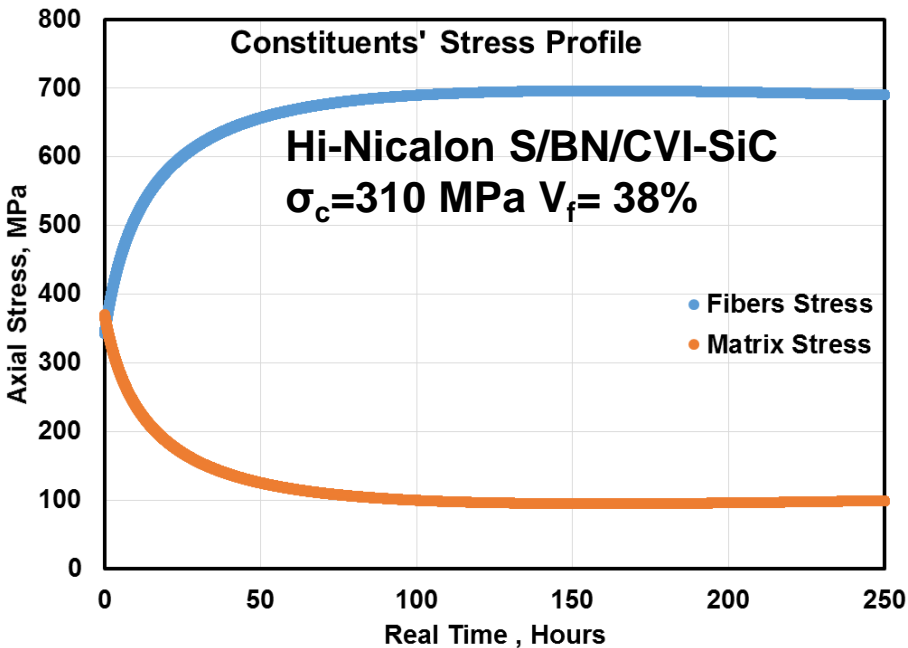
Creep Model Results for Constant Composite Stress & Different Fiber Content



Creep Model Results for Different Composite Stress & Different Fiber Content



Creep Model Results for Precracked Hi-Nicalon S Minicomposite



- Assumed extreme average fiber loading for precracked Hi-Nicalon S/BN/CVI-SiC (HNSPC1-5) $\sigma_c = 310 \text{ MPa}$ $V_f = 38\%$ $t_r = 470 \text{ hours}$.
- Hi-Nicalon S fibers creep parameters in air were input to the creep model to study upper extreme creep condition.
- Overall model results overestimates creep strain but model evolution agrees with experimental data.
- This is due to lower stresses on the fibers in the debond region and regions where CVI-SiC is still intact.

Conclusions

- CVI-SiC creep properties at 1200°C determined.
- Derivation of creep equation generated a stress redistribution model between fibers and matrix in creep and helped to understand the effect of fiber content change on creep load sharing behavior.
- Load sharing model helped in constructing minicomposites' creep model.
- Similar model methodology can be used to predict/model true creep evolution within the same constituent (Oxidizing fibers).
- Single Hi-Nicalon S fibers creep data were compared to precracked minicomposites data with the same fiber type.

